

APPENDIX G

Land Crab Exoskeleton and Tissue-Only Sample Analysis

Ridolfi identified discrepancies in the original Vieques Island land crab exoskeleton data received from Columbia Analytical Services (CAS) in September 2005. Specifically, calcium results from one analytical batch (laboratory service request [LSR] K0501162) were an order of magnitude lower than would be expected from samples of crab shell. In response, CAS re-analyzed the exoskeleton samples, which had been archived separately from the tissue at the laboratory at -20° C. Select exoskeleton samples from a second analytical batch (LSR K0501249) were also reanalyzed to verify those calcium results, which appeared to be within the appropriate concentration range.

Visual examination and comparison of laboratory-archived tissue materials, both homogenized and freeze-dried, for LSR K0501162 strongly suggested that the freeze-dried material analyzed by CAS was not the exoskeleton portion, as specified. Based on the color and texture of the freeze-dried material, the homogenized (and analyzed) sample was suspected of consisting of the internal tissue materials that remained after the shell was removed.

A total of six exoskeleton samples from LSR K0501162 were reanalyzed (Table G-1). Results confirmed that much higher calcium values were characteristic of the exoskeleton and that the laboratory's original results most likely reflected inadvertent analysis of the crab tissue rather than the shell. Thus, the exoskeleton reanalysis results for these six samples *replace* the original data reported by the laboratory.

Three exoskeleton samples from LSR K0501249 were also reanalyzed (Table G-1). Results were comparable to the results originally reported for these samples, confirming appropriate analysis of exoskeleton in the original batch. These analytical results are considered duplicates and were used only for purposes of data evaluation.

In the process of reanalyzing the exoskeleton samples, the corresponding tissue samples were also analyzed to better evaluate preferential accumulation of metals. These tissues had not been previously analyzed. Thus, one land crab specimen from each of the 14 locations was analyzed for metals in the tissue as well as in the exoskeleton.

In summary, the following exoskeleton and tissue samples, presented by LSR number, were analyzed by the laboratory in January 2006 (see notes below).

Table G-1. Exoskeleton and Tissue Sample Analysis, January 2006

Original LSR	Area	Sample ID	Exoskeleton	Tissue
K0501249	Area 1	S7-LC-01-01	-	c
K0501249	Area 3	KA-LC-01-02	a	c
K0501249	Area 4	LA-LC-01-01	a	c
K0501249	Area 5	S4-LC-01-03	-	c
K0501249	Area 7	SB-LC-01-04	a	c
K0501249	Area 12	LI-LC-01-02	-	c
K0501249	Area 13	VR-LC-01-03	-	c
K0501162	Area 2	JR-LC-01-02	b	c
K0501162	Area 6	PG-LC-01-06	b	c
K0501162	Area 8	PF-LC-01-01	b	c
K0501162	Area 9	RB-LC-01-06	b	c
K0501162	Area 10	BB-LC-01-04	b	c
K0501162	Area 11	BT-LC-01-03	b	c
K0501711	Area 14	HR-05	-	c

Notes:

- a. Reanalysis confirmed the original data are correct and usable (new data are considered duplicates).
- b. Reanalysis confirmed the original data represented an error in sample preparation; new data replaced original data.
- c. New analysis of tissue-only samples will be used for comparison with exoskeleton data.
- Original data determined to be correct based on “a” above.

Results from the reanalyzed exoskeleton samples confirmed that the original data reported under LRS K0501162 did not represent the exoskeleton materials. The data presented in this report reflect the results of the analysis of exoskeleton material, as well as the remaining tissue, conducted in January 2006 (under new LSR numbers K056373 and K056383). Results of the reanalysis of exoskeleton samples originally reported under LRS K0501249 confirmed the original results; therefore, the original values were retained.

This effort verifies that the sample processing error was associated only with LRS K0501162. In addition, corresponding tissue-only samples were analyzed to allow a direct comparison with exoskeleton samples in an evaluation of metal accumulation issues.